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Effects of curing methods and temperatures on mechanical properties of reactive powder concretes

Efeitos dos métodos de cura e temperaturas nas propriedades mecânicas de concretos de pós reativos

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Abstract

Reactive Powder Concretes are currently one of the most advanced cementitious composites presenting high mechanical resistance combined with a dense and modified microstructure compared to an ordinary concrete. This high performance is obtained throught the curing process, employing thermal treatment, aiming to enhance the pozzolanic reactions of the constituents present in these concretes, promoting advantageous changes in the microstructure of the material. This research evaluated the influence of different thermal curing methods (conventional, submerged, steamed, and hot air) under different temperatures (23°C, 50°C, 70°C and 90°C) on the compressive strength of a Reactive Powder Concrete, resulting variances over 70% of compressive strength between different curing methods, where it was concluded that steam curing method is the most efficient. The curing temperature of 90°C resulted in the highest values of compressive strength for the tested specimens.
Keywords: Reactive Powder Concretes. Thermal Curing. Compressive Strength. Mechanical Properties.

Resumo

Concretos de pós reativos são um dos compósitos cimentícios mais avançados apresentando alta resistência mecânica combinada com uma densa e modificada microestrutura comparada a um concreto ordinário. Este elevado desempenho é obtido através do processo de cura, empregando tratamento térmico, que tem como finalidade potencializar as reações pozolânicas dos constituintes presentes neste concreto, promovendo mudanças vantajosas na microestrutura do material. Esta pesquisa avaliou a influência de diferentes métodos de cura térmica (convencional, submersa, a vapor e ao ar quente) sob diferentes temperaturas (23°C, 50°C, 70°C e 90°C) na resistência a compressão de concretos de pós reativos, resultando em uma variação superior a 70% na resistência à compressão entre diferentes métodos de cura, onde foi concluído que a cura pelo método a vapor é a mais eficiente. A temperatura de cura de 90°C resultou nos maiores valores de compressão para os corpos de prova testados.
Palavras-chave: Concretos de Pós Reativos. Cura Térmica. Resistência à Compressão. Propriedades Mecânicas.

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Introduction

An ordinary concrete is a material basically composed of a binder medium in which the aggregate particles are agglutinated. The sand and gravel constitute the aggregates of this mixture and cement and water create an agglomerating medium (MEHTA; MONTEIRO, 2006; NEVILLE; BROOKS, 2010). The great acceptance of concrete, as a structural material, goes beyond its mechanical properties, since aspects such as fire and water penetration resistances are also important to place it as one of the most useful materials for structural purposes in Civil Engineering (MEHTA; MONTEIRO, 2006). Concrete is the most widely used construction material in the world for the execution of structures in Civil Engineering. However, the limitations related to the conventional use of this material, due to its low tensile strength and almost no ductility, present a difficulty to be overcome through studies in the field of Civil Engineering. In this way, Reactive Powder Concretes (RPC) turns out to be an attractive material capable of satisfying these limitations presented by the conventional concrete (YANG; JOH; KIM, 2010).

The development of high performance materials for structural applications in Civil Engineering has grown significantly in recent years (EL-HACHA; CHEN, 2012). In fact, RPC are presented as one of the most advanced cementitious composites in the field of Civil Engineering, bearing in mind that the development of RPC was possible due to the progress in the materials technology of its constituents (ZBED, 2013). RPC are the result of researches that aimed at concretes with superior characteristics, if compared to conventional ones, mainly in relation to their mechanical properties, thus allowing executing secure slender structural elements. In addition, favoring the use of slender structures, there is a lower consumption of natural raw materials and, consequently, a lower extraction of these resources (YI et al., 2012; ZBED, 2013).

RPC is defined as belonging to the family of ultra high performance concretes that use low water / agglomerant ratios and have uniaxial compression strength capable of exceeding 150 MPa, being basically constituted of: Portland cement, silica fume, quartz powder, quartz sand, water and superplasticizer. In addition, the RPC can be reinforced with metallic or polymeric fibers in order to improve the ductility of concrete. Thus, in terms of mechanical strength, RPC can reach from 3 to 16 times the compressive strength of a conventional concrete and up to 7 times the tensile strength, depending on the used materials and methods in its elaboration (WILLE; NAAMAN; PARRA-MONTESINOS, 2011; WANG et al., 2015).

According to (RICHARD; CHEYREZI, 1995), the first researches about RPC were credited to Bouygues from 1990 to 1995. Currently, several lines of research dealing with RPC are available in the literature, where significant articles can be found regarding: The best physical arrangement of the particles, defined as packing particles (LARRARD; SEDRAN, 1994; BONNEAU et al., 2000), the influence of different cement types (DILS; BOEL; SCHUTTER, 2013; ALKAYSI et al., 2016; WANG et al., 2017), mineral admixtures (RONG et al., 2015; SOLIMAN; TAGNIT-HAMOU, 2017), and superplasticizer (SCHÖFL; GRUBER; PLANK, 2012), the use of steel or polymeric fibers (SU et al., 2016; ZHOU; UCHIDA, 2017), the curing methodology and its influence on mechanical properties of RPC (KODUR et al., 2016; MOSTOFINEJAD; NIKOO; HOSSEIN, 2016), rheological properties (CHOI et al., 2016) and durability aspects (GHAFARI et al., 2015; TAFRAOUI; ESCADEILLAS; VIDAL, 2016).

RPC assumes as an axiom to be a material with a minimum defects, such as pores and microcracks. In this way, the main aspects related to obtaining a superior mechanical strength aim at minimizing porosity, modifying the microstructure of the cementitious matrix and increasing the physical homogeneity of its constituents. In this context, concerning the matrix structure modification, the curing procedure with the adoption of thermal treatment aims to potentialize the pozzolanic activity, mainly the silica fume, promoting a modification on the concrete microstructure, which leads to an improvement on its mechanical properties (ZBED, 2013; WANG et al., 2015).

According to micrographics and mineralogical analyzes by X-Ray Diffraction, presented in the literature, the curing process under thermal treatment creates a favorable environment for the pozzolanic reactions, where the silica fume consumes the crystals of Calcium Hydroxide ($Ca(OH)_2$) resulting in the formation of Calcium Silicate Hydrated (C-S-H), which is the main responsible for the mechanical strength of concrete (CHEYREZY; MARET; FROUIN, 1995; REDA; SHRIVE; GILLOT, 1999; AHMAD; AZAD; HAKEEM, 2014). In this way, a recently research confirmed the formation of secondary hydration products, under different cure methodologies, through X-Ray Diffraction and spectroscopy analysis, where the presence of products such as tobermorite and xonotlite were verified (HIREMATH; YARAGAL, 2017).

Regarding the curing process, the adoption of thermal treatments in early ages refers to provide benefits on the microstructure of RPC. Generally, thermal curing procedure consists in subjecting the specimens to a heat treatment for a period of 48 to 72 hours and, after this exposure, specimens are cured at the temperature of 23 ± 2°C in a humid chamber (ZBED, 2013; HASSAN; JONES; MAHMUD, 2012; AZAD; HAKEEM, 2013).

There are many works about thermal curing methods (TAI; PAN; KUNG, 2011; AHMAD; AZAD; HAKEEM, 2014; PREM; BHARATKUMAR; IYER, 2013; KODUR et al., 2016; MOSTOFINEJAD; NIKOO; HOSSEIN, 2016; WU; SHI; HE, 2017), however, a significant part of these researches present curing temperatures above 200°C and doesn't describe about the methods used to preserve a minimum relative humidity of 95% during the curing process. It is clear that for practical applications, as in a precast manufacteres of concrete structural elements, the maintence of a minimum relative humidity of 95% together with high temperatures for curing process could be difficult. In this sense, the aim of this work is to discuss the influence of curing methods and temperature on the compressive strength of RPC using four different curing methods (conventional, submerged, steam and hot air) and four different temperatures up to 100°C (23°C, 50°C, 70°C and 90°C) respecting a minimum relative humidity of 95% in the conventional, submerged and steam methods. The motivation for this study is related to the knowledge of the strength evolution in RPC by means of the thermal curing treatment to be applied in the civil construction industry in order to determine the most suitable relationship between the method and the curing temperature.

Experimental program

The materials used in this work are commonly commercialized in Brazil. The Portland cement CPV-ARI (equivalent to ASTM Type III), quartz sand with a maximum diameter of 600 μm and specific massa of 2.60 g/cm^3, quartz powder with a maximum diameter of 25 μm and specific mass of 2.65 g/cm^3, silica fume with a maximum diameter of 43 μm and specific mass od 2.15 g/cm^3, and commercial Polycarboxylate Ether superplasticizer were used. The chemical composition of the cement is presented in Table 1 according to Brazilian standard (ABNT, 1991), while Tables 2 and 3 present, respectively, the chemical composition of the silica fume and quartz sand determined by X-ray fluorescence spectroscopy.

At the first part of this research, a study of the constituent proportion of RPC was carried out in the laboratory with the available materials. The objective was to determinate the proportion of materials to be used for the production of a RPC in order to identify the best packing particles and the highest compression strength. Table 4

Table 1 – Chemical characteristics of Portland cement CPV-ARI, %.

Clinker and Calcium Sulphate	Carbon Dioxide	Loss Ignition	Insoluble Residue
95.0	3.0	4.5	1.0

Fonte: The author.

Table 2 – Chemical characteristics of silica fume, %.

SiO_2	Fe_2O_3	Al_2O_3	TiO_2	Loss Ignition
99.68	0.045	0.23	0.028	0.11

Fonte: The author.

Table 3 – Chemical characteristics of quartz sand, %.

SiO_2	Loss Ignition
99.62	0.07

Fonte: The author.

presents the final materials proportion used in this work. After this, a total of 60 specimens with a diameter of 5 cm and a height of 10 cm were molded and subsequently subjected to different curing methods and temperatures up to the date of compression test (28 days).

During the second part of this research, a thermal curing chamber was constructed in order to promote the thermal curing under steam and submerged conditions for different controlled temperatures. The equipment is a thermal insulating compartment with lid and a device to control the temperature of the water inside the compartment. In this way, it is possible to heat the water and generate steam inside the compartment controlling the environment temperature. A thermometer was coupled in the top of the compartment in order to measure and control the internal temperature. The temperature control was done by a central with precision of 0.1°C and reaches temperatures up to 120°C. Temperature and watertightness tests of the compartment were carried out before exposing the specimens attesting its efficiency. The equipment is presented on Figure 1 and a schematic representation of the equipment is presented on Figure 2.

Four curing methods, under different temperatures, were adopted in this work:

- Method A - Conventional cure in a humid chamber at 23 ± 2°C (reference condition);
- Method B - Thermal cure in hot air in a laboratory stove at 50°C, 70°C and 90°C;
- Method C - Thermal steam cure in the thermal curing

Table 4 – Materials proportion of RPC, in mass.

Cement	Quartz sand	Silica fume	Quartz powder	Superplasticizer	Water
1	1.44	0.34	0.3	0.03	0.33

Fonte: The author.

Figure 1 – Equipment developed for thermal curing.

Fonte: The author.

chamber developed at 50°C, 70°C and 90°C;

- Method D - Submerged thermal cure in the thermal curing chamber developed at 50°C, 70°C and 90°C.

The thermal cure temperatures of 90°C was defined as the maximum curing temperature of the specimens, since, according to (CHEYREZY; MARET; FROUIN, 1995), more than 90% of the pozzolanic activity occurs at this temperature. The temperature of 50°C was defined as the lowest possible temperature for steam generation inside the thermal chamber, according to preliminary tests. The temperature of 70°C was defined as an intermediate temperature to be evaluated between the maximum and minimum temperatures used in this work. The temperature inside the humid chamber, for conventional curing method, ranges from 21 to 25°C (ABNT, 2015) under laboratorial conditions, this was considered as the reference condition.

In order to identify the method and curing temperature of the six specimens used in each group, the nomenclature adopted corresponds to the letter identifying the curing method followed by the curing temperature applied, as shown in Table5. Thus, the group identified as B70, for example, corresponds to the group of hot air-cured specimens (Method B) at a temperature of 70°C.

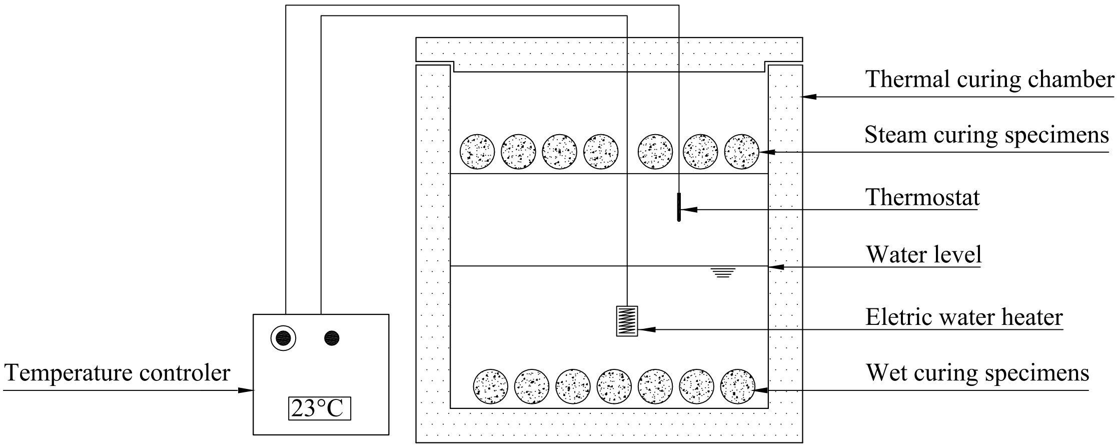
The concrete was produced in a planetary mixer where dry materials were placed in the mixer and homogenized for 5 minutes. In sequence, the water was added with the superplasticizer and the mix continued for another 10 minutes, observing the homogeneity of the concrete. After mixing the materials, the molds of the specimens were filled, covered by a protective film (in order to prevent loss of water), and left for 24 hours at laboratory temperature (23°C). After this, then were demoulded, identified and sent to their respective curing condition. The specimens subjected to the thermal curing treatments remained for 72 h under their respectives conditions and temperatures. Subsequently, the specimens were moved to the humid chamber in order to cure in a conventional condition until the date defined for the compression test (28 days).

Results and discussion

Table 6 presents the average compressive strength and the standard deviation obtained for each group of specimens according to their curing conditions and temperatures. Figure 3 graphically shows the average compressive strengths obtained for specimens subjected to thermal curing conditions.

Comparing the results of average compressive strength, it is possible, in a first analysis, to observe that curing method and temperature have a great influence on the mechanical strength of RPC. The results presented in Table 6 and in Figure 3 show that, regardless of the curing temperature, the groups belonging to method B presented a lower compressive strength in comparison of the compressive strength obtained by conventionally cured specimens without heat treatment. In this sense, specimens belonging to the B50 group had an average compressive strength of 21.54% lower than the referenc e test specimens (group A23). The average compressive strength of B70 and B90 groups were 16.13% and 1.59% lower than the average compressive strength of group A23 respectively. This fact is related to the humidity of the hot air curing environment, where it was not possible to ensure a minimum relative humidity of 95% for the curing environment, demonstrating that not only the temperature, but also moisture conditions during the curing process are essential for the development of hydration and pozzolanic reactions in order to improve the performance of RPC. In this way, it is possible to conclude that the non-compliance of curing procedures aimed to ensure a minimum relative humidity of 95% can difficult the development of pozzolanic reactions and, at

Figure 2 – Schematic representation of the equipment developed.



Fonte: The author.

Table 5 – Nomenclature used to identify the curing methods groups.

Identification	Number of Specimens	Curing method and temperature
A23	6	Conventional curing in humid chamber at 23°C and minimum relative humidity of 95% (reference condition)
B50	6	Thermal hot air curing at 50°C and relative humidity below 95%
B70	6	Thermal hot air curing at 70°C and relative humidity below 95%
B90	6	Thermal hot air curing at 90°C and relative humidity below 95%
C50	6	Thermal steam curing at 50°C and minimum relative humidity of 95%
C70	6	Thermal steam curing at 70°C and minimum relative humidity of 95%
C90	6	Thermal steam curing at 90°C and minimum relative humidity of 95%
D50	6	Thermal submerged curing at 50°C and minimum relative humidity of 95%
D70	6	Thermal submerged curing at 70°C and minimum relative humidity of 95%
D90	6	Thermal submerged curing at 90°C and minimum relative humidity of 95%

Fonte: The author.

Table 6 – Average compressive strengths results (MPa).

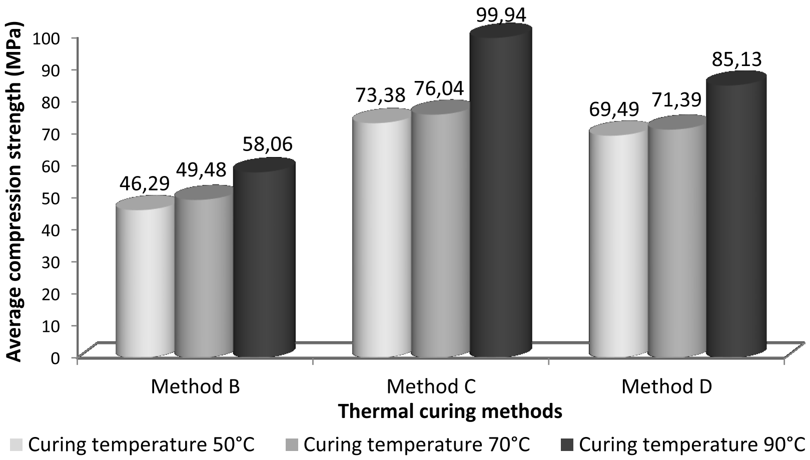
Curing Method	A23	B50	B70	B90	C50	C70	C90	D50	D70	D90
Average compressive strength (MPa)	59	46.29	49.48	58.06	73.38	76.04	99.94	69.49	71.39	85.13
Standard deviation (MPa)	3.27	8.45	4.36	1.69	8.78	1.41	3.58	9.42	2.85	3.63

Fonte: The author.

the same time, affect the mechanical strength of RPC. In this sense, Table 7 shows the increase or decrease percentage of the average compressive strength relative to the reference group (A23), where it is observed that only method B presented a decrease in relation to the average compressive strength of the group A23 (59 MPa), consequently, this method is not recommended for practical applications.

The results presented on Table 6 and Figure 3, for different curing methods show that, in all temperatures, higher average compressive strength was achieved by the steam curing condition (Method C) and the lowest was observed using the hot air curing condition (Method B). The submerged curing condition (method D) presented average

Figure 3 – Average compressive strength according to the curing method and temperature.



Fonte: The author.

Table 7 – Percentage relation between the average compressive strength of each thermal curing group relative to the reference group (A23).

	Curing temperature		
Curing Method	50°C	70°C	90°C
Method B	-21.54%	-16.13%	-1.59%
Method C	24.37%	28.88%	69.39%
Method D	17.78%	21.00%	44.29%

Fonte: The author.

strength close to the obtained by the steam condition, but always lowers. It is observed that an increase in the curing temperature from 70°C to 90°C, under regimes that ensure a minimum relative humidity of 95% (methods C and D), a significant increase in the average compressive strength was achieved, as it is possible to note comparing the mean strengths in C70 and C90 and also D70 and D90 in Table 6. On the other hand, comparing the increase of compressive strength at temperatures of 50 and 70°C under the same conditions, it is observed that the strength increase is substantial, according comparison between the average compressive strength obtained in conditions C50 and C70 and D50 and D70 in Table 6. This fact demonstrates that pozzolanic activity in RPC is significantly potentialized at temperatures of 90°C, in agreement with the literature (CHEYREZY; MARET; FROUIN, 1995), where the authors affirm that at this temperature the pozzolanic activity reaches values of the order of 90%.

In this sense, Table 8 presents the percentage relation between the average compressive strengths according to different temperatures, where it is possible to note that, in percentage terms, the increase of mechanical strength by raising the cure temperature from 50°C to 70°C is limited to values between 2 and 7%, while for 50°C to 90°C the gain overcome 36% for the methods employed in this research. Consequently, the increase in compressive strength becomes significant, in fact, only when the temperatures reach levels of the order of 90°C.

Table 8 – Relation between average compressive strengths according to the curing temperatures (%).

	Curing temperature		
Curing Method	50°C/70°C	50°C/90°C	70°C/90°C
Method B	6.89%	25.42%	17.34%
Method C	3.63%	36.19%	31.43%
Method D	2.73%	22.50%	19.24%

Fonte: The author.

Figure 4 presents a comparative analysis between the results of average compressive strength obtained according to different curing methods and temperatures, while Table 9 presents the percentage variation of strength variation according to temperature.

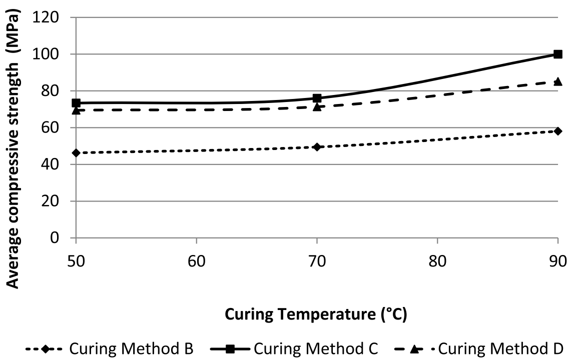
Table 9 – Relation between average compressive strengths according to the curing method (%).

	Compressive strength variation according to the curing method		
Temperature (°C)	B/C	B/D	C/D
50	58.51	50.11	5.59
70	53.67	44.27	6.52
90	72.14	46.63	17.39

Fonte: The author.

In analysis from Figure 4 it is possible to note that thermal steam cure proved to be the most efficient in terms of compressive strength for RPC, followed by the sub-

Figure 4 – Comparative results of average compressive strength between different curing methods.



Fonte: The author.

merged curing condition and the hot air curing condition, respectively. Moreover, in all cases, the curves show a similar increase of average compressive strength, where it is observed that a significant increase in the curve gradient occurs between the temperatures of 70 and 90°C. In addition, in Figure 3 it is possible to note that the average compressive strengths are similar between curing methods C and D for temperatures of 50 and 70°C, showing that specimens cured at temperatures limited to 70°C, either by the submerged or steam method, the mechanical behavior relative to the compression strength is similar.

In fact, Table 9 shows that the resistance variation between methods C and D for temperatures of 50° C and 70°C is less than 7%. In Table 9 it is still possible to highlight the effects of different cure methods, where it is observed that at 90°C the difference between the compressive strength values between methods B and C exceeded 70%, demonstrating again the importance of maintaining a minimum relative humidity of 95% during the curing process, and confirming that, for practical applications that steam cure at 90°C is the most appropriated.

Conclusions

- For hot air curing method, the results of compressive strength were lower than the values obtained for specimens subjected to conventional curing method in a humid chamber demonstrating that no only the temperature, but also the maintenance of a minimum relative humidity of 95% is primordial for the effectiveness of the curing process in order to potentialize the pozzolanic reactions.
- The steam curing method proved to be the most efficient, leading to the highest compressive strength at all observed temperatures. This fact is related to the maintenance of a minimum relative humidity of 95% in the curing environment during the heating process. Moreover, at a temperature of 90°C, the percentual difference in compressive strength between methods that promote the maintenance of the relative humidity of the curing environment and the method that does not promote such maintenance reached values higher than 70%.
- A growing trend of compressive strength was observed as the temperature is increased for all the curing methods applied in this work. In this case, regardless of the curing method, the highest compressive strengths were reached at 90°C, fact this related to the potentialization of pozzolanic reactions in RPC. Thus, the steam curing method at a temperature of 90°C proved to be the most suitable for practical applications on the Civil Construction industry.
- Curing temperatures below 70°C did not present significant increases in compressive strength for all applied methods, even ensuring a minimum relative humidity of 95% during the curing process. The highest strength gradiente was observed when the curing temperature increase from 70°C to 90°C, where variations greater than 30% were observed.

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